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Blue LED-Driven Synthesis of Benzoyl-Guanidine Hybrids *via* Radical-Radical Cross-Coupling

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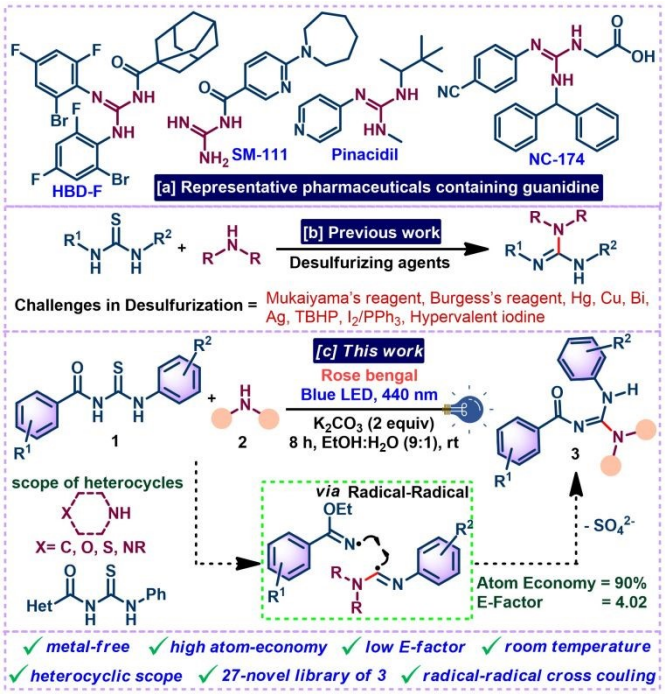
We describe a radical-radical coupling strategy for guanylation of benzoyl-thiourea derivatives, with diverse secondary amines to afford bioactive benzoyl-guanidine hybrids under blue LED irradiation in a greener reaction media. Moreover, this effective, high atom-economy (90%) protocol involves molecular cleavage (*via* Mumm rearrangement), formation of revised fragments, and reassembly of selective radical fragments. In addition, the work demonstrates postmodification of the benzoyl guanidine to bioactive isoquinolinone scaffolds *via* ruthenium-catalyzed oxidative annulation with diphenylacetylene.

The commanding influence of amine functionality on the small molecules can significantly tune the pharmacokinetic and bioactivity of the functional molecules.[1a] Hence, the inclusion of such functionalities has foremost importance in the synthetic organic chemistry and chemical biology. In this regard, guanidine, a *N*-enriched bioactive hybrid, has received considerable attention in the pharmaceutical and functional heterocycles due to its well-known bioactivity[1b-c] such as anti-bacterial,[2a] anti-fungal,[2a] anti-protozoal,[2b] anti-tumor,[2c] and anti-leishmanial activity.[2d] Whereas, guanidine-based drug moieties, including pinacidil (treatment of hypertension),[3a] NC-147 (synthetic sweetener),[3b] HBD-F (anti-bacterial),[3c] and SM-111[3d] (anti-HIV agent) have brought them to the forefront of research (Scheme 1a). These compounds are not only biologically significant but also important intermediates in synthetic chemistry.[4]

Moreover, the strong basicity of guanidine makes it an efficient organic superbase, enabling its application in both stoichiometric and catalytic organic transformations.[5] Additionally, guanidines are also used as chiral auxiliaries and chiral Bronsted base organo-catalysts in numerous asymmetric synthesis, including Mannich-, Michael-, and Henry- type reactions.[6] However, the synthetic routes for guanylation are



Scheme 1. Synthetic exploration of bioactive benzoyl-guanidine

limited and suffer from several disadvantages, such as the use of stoichiometric oxidants, metals, and desulfurizing agents (Scheme 1b).[7] To promote guanylation mainly requires guanylating precursors like carbodiimide,[8a] isothiourea,[8b] triflylguanidines,[8c] cyanamide,[8d] amidine,[8e] pyrazole-1-carboximidamides,[8f] benzoyl-thioureas.[8g] In the previously documented methodologies, Wacharasindhu *et al.* in 2020 reported a $Ru(bpy)_3Cl_2$ photocatalytic approach to access guanidine.[9a] However, the synthesis of benzoyl-guanidine possesses several challenges, yet seminal reports from Badshah *et al.* (2012)[9b] and Saeed *et al.* (2025)[9c] notably utilized highly toxic $HgCl_2$ in their respective preparation. Besides, Brito group in 2015 and Tamminana *et al*. in 2021 reported the synthesis of benzoyl-guanidine from benzoyl-thiourea and amines using an oxidant such as TBHP and iodine (50 mol%), respectively (Scheme 1b).[9d-e]

Driven by growing demand for sustainable[10] and energy-efficient processes, [11] photoredox chemistry has emerged as a

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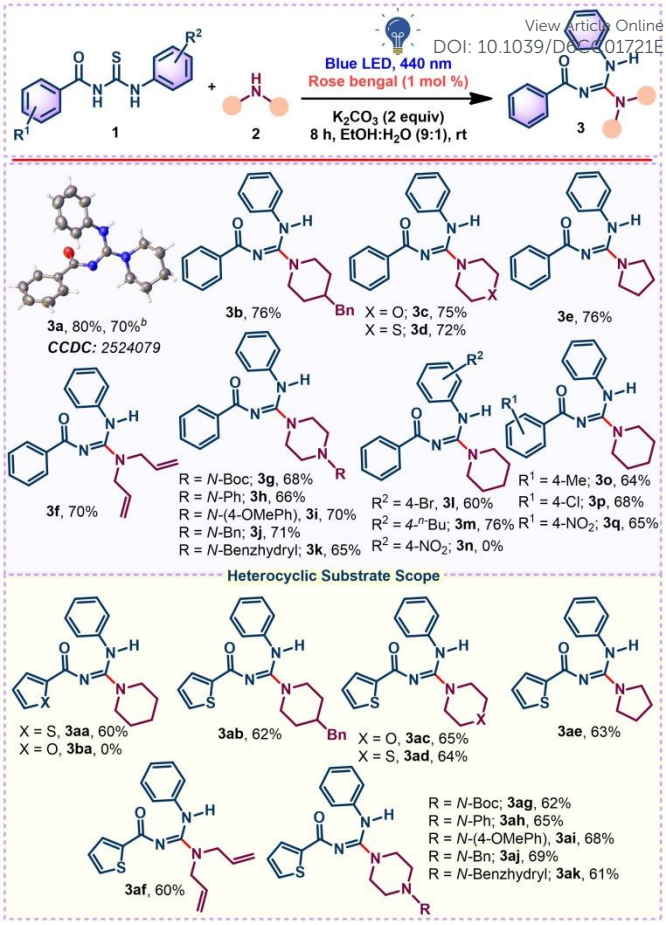
powerful platform for the rapid organic transformations under mild conditions.[12] Although rearrangement reactions involving bond cleavage and reconstruction offer atom-economical synthetic routes,[13a-c] many reported methods suffer from poor efficiency, are not selective, and result in loss of components during fragmentation.[13d] To address these limitations and the longstanding desulfurization challenges in the efficient guanylation, we have introduced a mild metal-free photoredox electron transfer protocol in a green solvent under blue LED irradiation, which offers high atom-economy (90%).

The exploration commenced with a systematic optimization of the reaction parameters (Table S1-S5). Initially, we employed benzoyl-thiourea **1a** and piperidine **2a** as model substrates. The reaction was carried out in acetonitrile solvent, base K_2CO_3, rose bengal (RB) as a photocatalyst under blue LED irradiation for 8 hours. Under these circumstances, the desired benzoyl-guanidine **3a** (CCDC no: 2524079) was observed with a commendable 65% yield. Further, screening of photocatalysts reveals that eosin-y, rhodamine-6g, and g-C_3N_4 remained inferior to RB and produced 20-60% yields, respectively (Table S1). Further, the systematic screening of solvents like polar, non-polar, and a mixture of solvents, EtOH/H_2O 9:1 was found to be the optimal solvent producing **3a** with 80% yield (Table S2). Subsequently, after screening various bases (Table S3), we found that the K_2CO_3/EtOH:H_2O (9:1) combination was the best condition by considering its low toxicity and cost-effectiveness. Additionally, the effect of light and the photocatalyst was studied (Table S4-S5). In the absence of blue LED irradiation and photocatalyst, the absence of guanylation emphasizes the importance of photonic activation during the course of excitation and the importance of the photocatalyst in the guanylation *via* the electron transfer process, respectively. Moreover, the role of oxygen in the reaction was investigated by carrying out the reaction under an Argon atmosphere. The suppressed yield of the desired product indicates the importance of oxygen in the photocatalytic cycle.

After the reaction condition being optimized, we expanded the scope of the reaction by testing diverse amines with **1a** under the optimized conditions. Secondary aliphatic amines such as 4-benzylpiperidine, morpholine, thiomorpholine, pyrrolidine, and allylamine were smoothly reacted with **1a** under optimized conditions, producing the respective benzoyl-guanidine **3b-3f** in high yield (70-76%). Further, the scope was extended with diverse piperazines, and the reaction yielded desired products in good yields. When *N*-Boc piperazine, *N*-phenyl piperazine, *N*-(4-methoxyphenyl)piperazine, *N*-benzyl piperazine, and *N*-benzhydryl piperazine were reacted with **1a**, the corresponding benzoyl-guanidine **3g-3k** were formed in moderate to good yield (65-71%). Next, we examined the effect of various substituents on **1a**. For instance, when benzoyl-thiourea containing halide (R^2 = 4-Br) was made to react with piperidine, the desired product **3l** was obtained with 60% yield. The decreasing yield might be due to the electron-withdrawing (EWG) inductive effect of Br, which reduces the nucleophilicity of the thiourea nitrogen.[14a] Similarly, an electron-donating



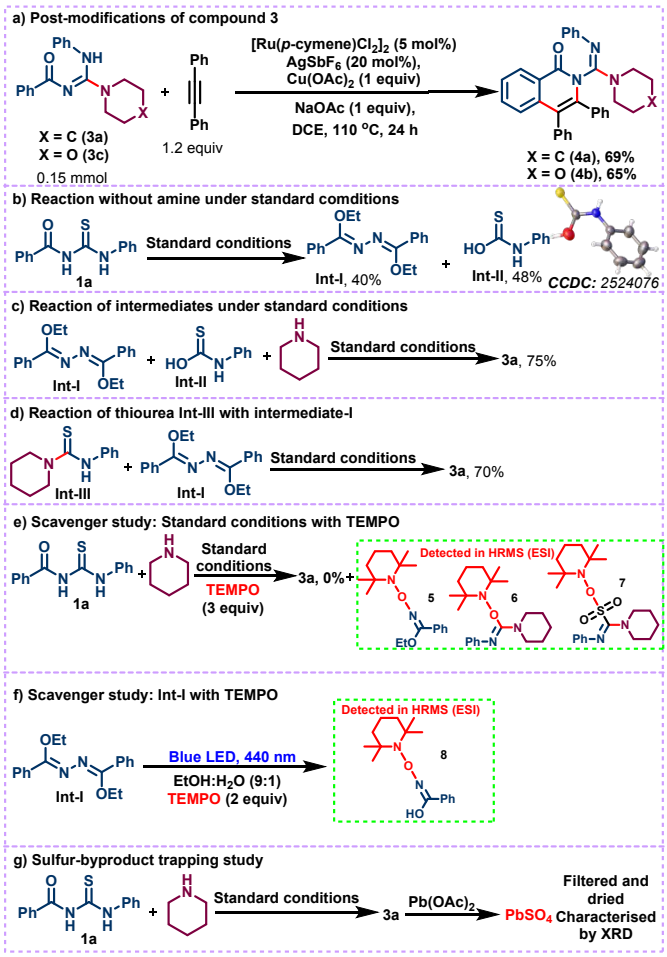
Scheme 2: Substrate scope. [a]Reaction conditions: **1a** (0.5 mmol), amine (1.5 equiv), K_2CO_3 (2 eq.), and rosebengal (1 mol%) in EtOH:H_2O (9:1, 4 mL) with blue LED irradiation at room temperature for 8 h. [b]Gram-scale synthesis (4 mmol).

group (EDG) like ^{n}butyl substituted benzoyl-thiourea (R^2 = 4-^{n}Bu) was made to react with piperidine to provide benzoyl-guanidine **3m** with good yield (76%). Unfortunately, a strong electron-withdrawing (R^2 = 4-NO_2) substituted benzoyl-thiourea failed to produce **3n**, likely due to the strong electron-withdrawing nature of the nitro group significantly decreases the electron density on the thiourea nitrogen and adjacent sulfur atom.[14b]

Moreover, the effect of substituents on the benzoyl side was also investigated, where R^1 = 4-Me, 4-Cl, 4-NO_2 substituted benzoyl-thiourea were smoothly reacted with piperidine, producing the compounds (**3o**-**3q**) with yields of 64-68%. After successful guanylation of benzoyl thiourea using secondary amines and piperazine derivatives, we further expanded the scope of the reaction with heterocyclic thiourea. When thiophenoyl-thiourea **1g**, and furanoyl-thiourea **1h** were made to react with piperidine, only thiophenoyl-thiourea was able to produce the desired product with 60% yield (**3aa**-**3ba**). This difference likely arises due to the higher oxidative sensitivity and low stability of the furan ring compared to thiophene under photoredox conditions, thereby making it more susceptible to oxidative decomposition.[14c-d] Further, the substrate scope was continued by reacting **1g** with various secondary amines such as 4-benzylpiperidine, morpholine, thiomorpholine, pyrrolidine, and allylamine, producing the respective benzoyl-guanidine

3ab-3af in moderate yield (60-65%). Further, the heterocyclic scope was extended with diverse piperazine derivatives. Where *N*-Boc piperazine, *N*-phenyl piperazine, *N*-(4-methoxyphenyl)piperazine, *N*-benzyl piperazine, and *N*-benzhydryl piperazine were reacted with **1g**, the desired heterocyclic-guanidine **3ag-3ak** with moderate to good yield (61-69%). Furthermore, we assessed the viability of our protocol for gram-scale reactions using 4 mmol of **1a** under standard conditions. The targeted product **3a** was observed with 70% yield, highlighting that the protocol was readily scalable. Moreover, the post-modifications of the obtained product were demonstrated through ruthenium-catalyzed oxidative annulation of compounds (**3a** and **3c**) with diphenylacetylene to afford bioactive isoquinolinone scaffolds (**4a -4b**) with 64-69% yield (Scheme 3a).[15]

To gain insight into the mechanism, a series of experiments was performed (Scheme 3b–3g). The **1a** was subjected to irradiation with blue LED under standard conditions for 8 hours in the absence of an amine source. Surprisingly, the starting material **1a** was consumed, and two different fragments, **Int-Ⅰ**[16] and **Int-Ⅱ** were observed, which were isolated and characterized by NMR and HRMS spectroscopy. Additionally, the crystal structure of **Int-Ⅱ** was obtained from SC-XRD (CCDC No:



Scheme 3. Control experiments.

2524076). Moreover, when the isolated fragments **Int-Ⅰ** and **Int-Ⅱ** were made to react with piperidine under standard conditions, the product **3a** was obtained with 75% yield. These

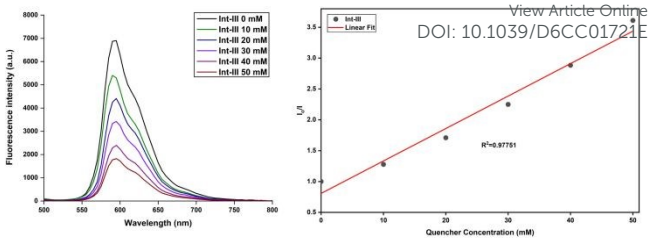
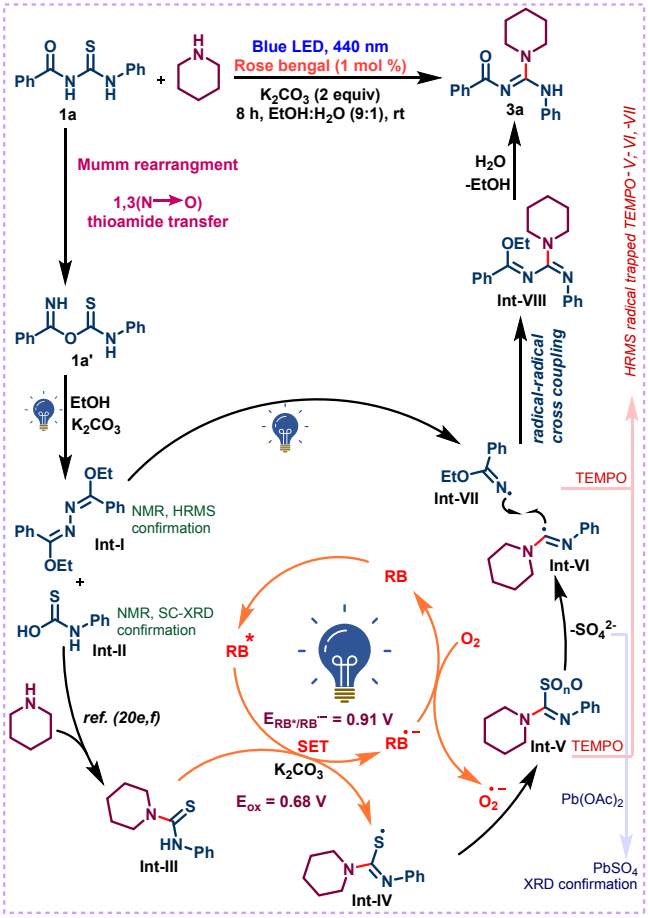


Figure 1. Fluorescence-quenching experiments, and Stern-Volmer plot.

findings suggested that **Int-Ⅰ** and **Int-Ⅱ** were the first key intermediates in this photoredox pathway. Next, the **Int-Ⅰ** was made to react with thiourea **Int-Ⅲ** under standard conditions, resulting in the formation of guanidine **3a** with 70%. Moreover, the investigation was also made on the radical scavenger experiment. When the reaction of **1a** and piperidine was carried out under standard condition in the presence of a radical scavenger (TEMPO), the yield of **3a** was suppressed. This confirmed that the reaction was proceeding through a radical pathway. Moreover, we succeeded in trapping the three crucial radical intermediates **5**, **6**, and **7** in the radical scavenger experiment, which was observed in the HRMS (Scheme 3e). Similarly, when the **Int-Ⅰ** was irradiated with blue LED in presence of TEMPO (2 equiv) in EtOH:H_2O, the radical intermediate **8** was observed in HRMS. Further, we succeeded in trapping the precipitate of sulfur byproduct after the addition of $Pb(OAc)_2$ in the reaction mixture (Scheme 3g). The trapping experiment was conducted in MeCN as a solvent, and DBU as a base to preclude the interference from solid K_2CO_3.[17] The resulting yellow precipitate was filtered and characterized by powder-XRD (Figure S5) and the resulting data corroborate that the byproduct was lead sulfate.

Moreover, UV-visible absorption spectroscopy of all compounds (**1a**, piperidine, K_2CO_3, **Int-Ⅰ**, **Int-Ⅱ**, RB) in EtOH:H_2O (9:1) was recorded (see Supporting Information). The observations reveal that electron transfer prevails rather than the energy transfer process, which was due to the absorption spectra of the individual molecules not overlapping with the emission spectrum of RB (Figure S8).[18] Further, we conducted Stern-Volmer fluorescence quenching experiments[18] in the presence of RB (Figure S11-S16). The results in figure 1 demonstrate that the excited state of RB* was majorly quenched by **Int-Ⅲ,** which assists the first reductive quenching cycle of RB*. Moreover, we measured the redox potential of **Int-Ⅲ**, and RB by cyclic voltammetry experiment[19] (see supporting information, figure S17-S18). From the experimental results, it was found that the oxidation potential of **Int-Ⅲ** E_{ox} = 0.68V vs Ag/AgCl and reduction potential of the excited state rose bengal (RB*) E_{red*} = 0.91V vs Ag/AgCl. These findings suggest that the **Int-Ⅲ** can participate in single electron transfer (SET) with RB*. Accordingly, it is reasonable to propose that RB* can oxidize **Int-Ⅲ** through a reductive quenching pathway.

Based on the results obtained from the above investigations, we proposed a mechanistic pathway. The reaction initiated with a Mumm-type rearrangement[20a-d] of **1a** to **1a'** intermediate, which was then fragmented into two separate intermediates (**Int-Ⅰ**, **Int-Ⅱ**) in the presence of base and ethanol. Further, the thioamidation[20e-f] of **Int-Ⅱ** to produce intermediate **Int-Ⅲ** (E_{ox}



Scheme 4. Proposed mechanism.

= 0.68V vs Ag/AgCl), which was later deprotonated by base and followed by SET with **RB*** ($E_{RB*/RB^{\bullet-}}$ = 0.91V vs Ag/AgCl)[21a-b] to generate radical intermediate **Int-Ⅳ**. Subsequently, **RB** was regenerated by a back single-electron-transfer (BET) from reduced state **RB**$^{\bullet-}$ to molecular oxygen O_2 (in air).[21c-d] This results in the formation of superoxide ($O_2^{\bullet-}$), which is further involved in the oxidative coupling with radical intermediate **Int-Ⅳ** to produce peroxysulfur intermediate **Int-Ⅴ**, followed by elimination of sulfate to produce the radical intermediate[22] **Int-Ⅵ**. Meanwhile, the intermediate **Int-Ⅰ** under irradiation with blue LED to produce radical intermediate **Int-Ⅶ**, which is further involved in radical-radical cross-coupling with radical intermediate **Int-Ⅵ** to generate intermediate **Int-Ⅷ**. Ultimately, hydrolysis of intermediate **Int-Ⅷ** to yield targeted benzoyl-guanidine **3a** (Scheme 4).

We have developed an efficient and sustainable approach to access benzoyl-guanidine hybrids *via* a radical-radical cross-coupling mechanism. Various derivatives of benzoyl-thiourea were treated with an array of secondary amines in the presence of rosebengal as a photocatalyst in EtOH: H_2O to develop a library of benzoyl-guanidine. The reaction proceeds *via* molecular cleavage (*via* Mumm-type rearrangement), fragments modification, and selective reassembly of radical fragments took place to offer benzoyl-guanidine with high atom-economy (90%).

Data availability

The data supporting this article has been included as part of supporting information (SI). Supporting Information: Experimental procedures, detailed information regarding optimization of the reaction conditions, detailed mechanistic investigations, characterization data of products, NMR (^{1}H, ^{13}C) spectral data, HRMS spectral data of new products, and X-ray crystallographic data of products.

Conflicts of interest

There are no conflicts to declare.

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Data availability

The data supporting this article has been included as part of supporting information (SI). Supporting Information: Experimental procedures, detailed information regarding optimization of the reaction conditions, detailed mechanistic investigations, characterization data of products, NMR (1H, 13C) spectral data, HRMS spectral data of new products, and X-ray crystallographic data of products.